User Guide for the
Philips CM200
FEG TEM

THE RULES:

1. If in doubt, **CLOSE THE GUN VALVE**.
2. Do not touch a control if you don't know exactly what it will do.
3. Never force anything beyond finger strength.
4. If in doubt, ask for help.

Contact staff for this instrument:
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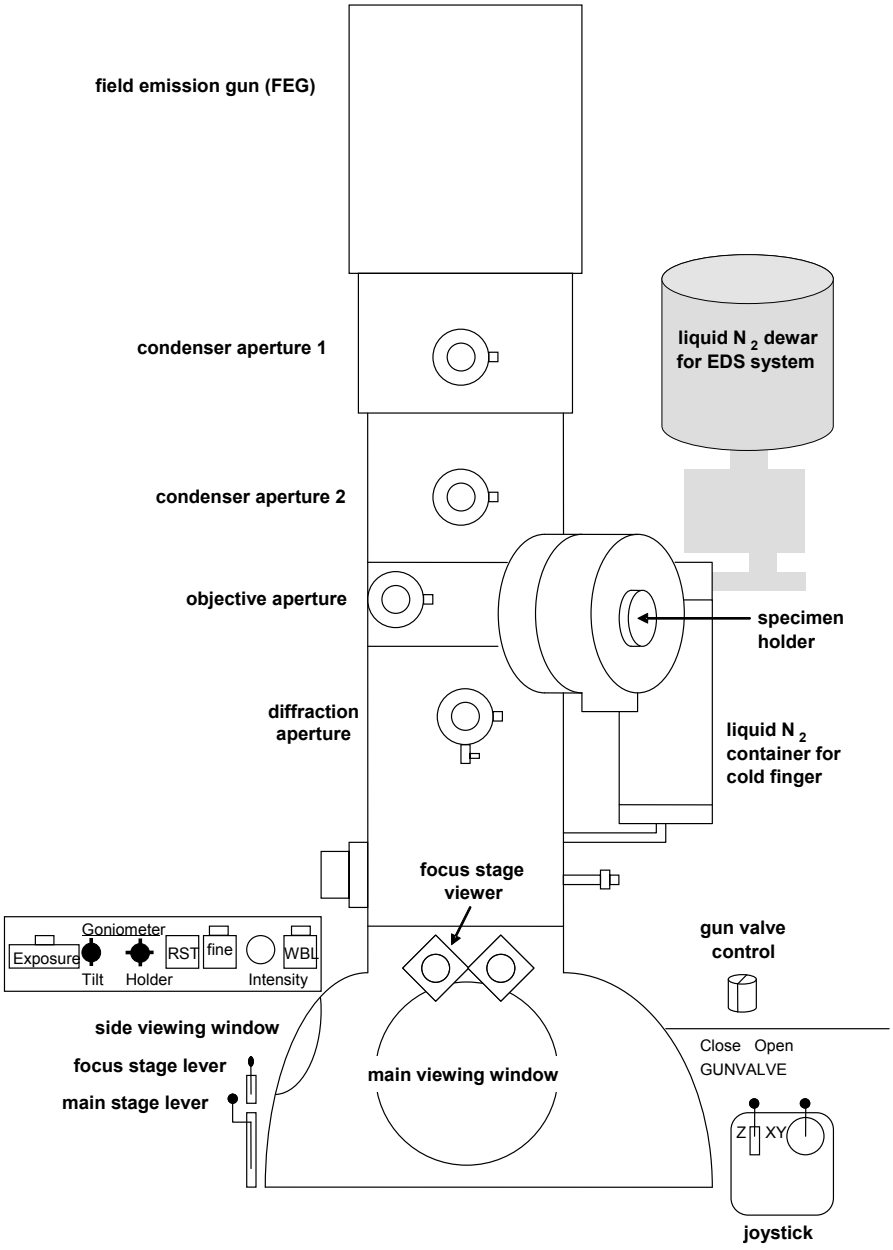
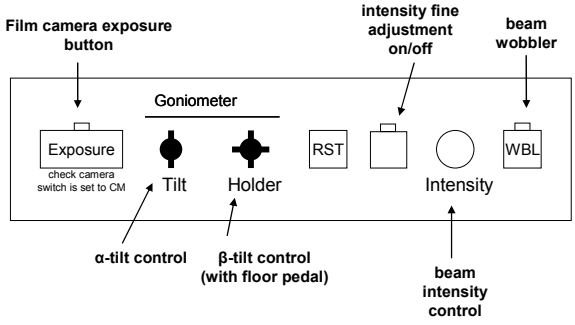
Contents

1. IMPORTANT PARTS OF THE PHILIPS CM200 TEM..... 4
2. CHECK cm200 STATUS 8
3. TURN THE FILAMENT UP 9
4. LOAD THE SPECIMEN 10
5. OBTAIN AN IMAGE 12
6. SET THE EUCENTRIC HEIGHT..... 13
7. FOCUS THE IMAGE..... 13
8. MOVE AROUND THE SPECIMEN 14
9. CHECK THE MINOR ALIGNMENTS 15
10. INSERT AN OBJECTIVE APERTURE..... 17
11. RECORD AN IMAGE ON PHOTOGRAPHIC FILM 18
12. RECORD A DIGITAL IMAGE 19
13. EXCHANGE SPECIMENS..... 20
14. END THE SESSION 21
Appendix a: Using the double tilt holder..... 22
Appendix b: Diffraction patterns 24
Appendix c: X-ray analysis 25
Appendix d: X-ray mapping/line scans 32

1. IMPORTANT PARTS OF THE PHILIPS CM200 TEM



To the left of the column:



The gun valve:

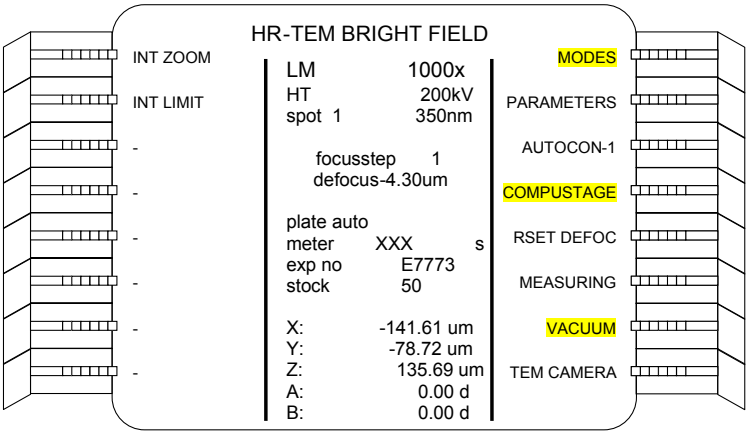
The Philips cm200 TEM uses a field emission gun (FEG) which provides very high resolution but requires more care than a standard TEM filament. It is very important that a high vacuum is maintained in the gun chamber at all times. The gun chamber is sealed off from the rest of the column using the gun valve. The gun valve **must** be closed whenever a specimen is being exchanged. If you have a problem with the operation of the instrument or if you need to leave the room, please close the gun valve by turning the control knob slowly clockwise to the closed position.

The computer screen:

The cm200 contains many manual controls, such as focus and magnification. However, many functions are controlled via a computer interface, using the "softkeys" at the edges of the screen. To select a particular function press the softkey next to the appropriate function title. To leave a page or function, press the READY button at the lower right hand side of the screen. In most cases, this will take you back to the HR-TEM BRIGHT FIELD page.

The functions described here are those required for most users. The other functions must not be used unless you have received appropriate training.

DO NOT PRESS A SOFTKEY IF YOU DON'T KNOW WHAT IT DOES.

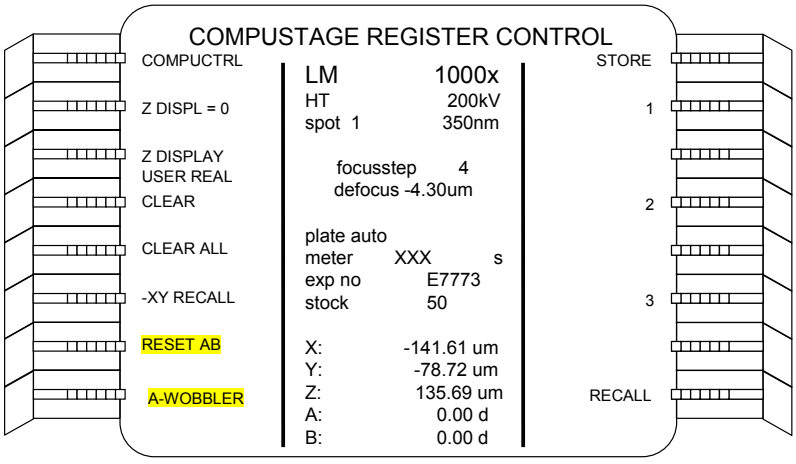


HR-TEM BRIGHT FIELD page

- This page allows access to most of the functions of the microscope.
- The page is made up of three columns. The central column shows the magnification, spotsize, focus step, film exposure information, film stock levels, and stage positions.
- The right-hand column allows access to other pages in the program via the softkeys. The most commonly required are COMPUSTAGE, VACUUM STATUS and MODE SELECTION (MODES).

COMPUSTAGE page

- The COMPUSTAGE allows stage position to be monitored and controlled. Stage positions can be stored and recalled. The central column on this page contains the same information as on the HR-TEM BRIGHT FIELD page.
- To store a stage position, select a number in the right-hand column on the monitor using a softkey (when selected, the number is highlighted). Press the STORE softkey to save the position.
- To return to a saved position, select the appropriate number and press the RECALL softkey on the right-hand side of the page. The stage will automatically move to that position.
- To clear a saved stage position, select the appropriate number and press the CLEAR softkey on the left-hand side of the page.
- To clear all saved stage positions, press the CLEAR ALL softkey *twice* (located on the left-hand side of the page).
- The COMPUSTAGE page also contains functions for setting eucentric height (A-WOBBLER) and for returning the specimen to zero tilt (RESET AB). The RESET AB softkey *must always be pressed* before a specimen is removed.



VACUUM STATUS page

- The VACUUM STATUS page shows a diagram of the vacuum system. The status of the vacuum can be read at the top of the screen. Below the diagram of the vacuum system is a series of pressure readings, including the IGP (ion getter pump) reading.

P1:	32	P3:	34
P2:	49	IGP:	5

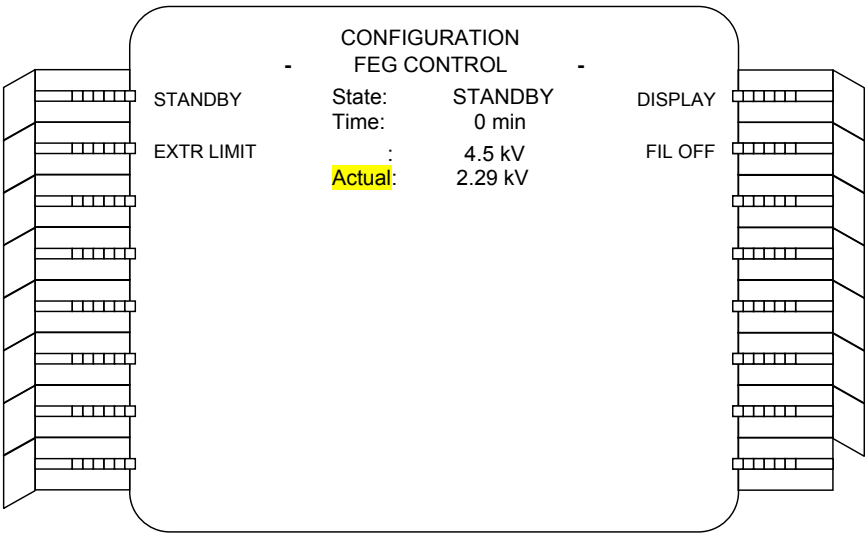
- Before the gun valve is opened, the IGP reading must be less than 27 and the VACUUM STATUS must be 'READY'.

MODE SELECTION page

- The modes page allows the user to select the TEM mode to be used. Usually this is HR-TEM. The CONFIGURATION page can be accessed from this page.

CONFIGURATION page

- This page contains information on the state of the microscope (STANDBY or OPERATE) and on the extraction voltage. As the filament is turned up, the change in extraction voltage ("Actual") can be monitored on this page.



2. CHECK cm200 STATUS

1. **Log in** to the EMU Booking System on the PC.

2. **Check** that the vacuum is OK by confirming that both the UHV and HV indicators on the far right side of the panel are illuminated. *If not, check with EMU staff.*

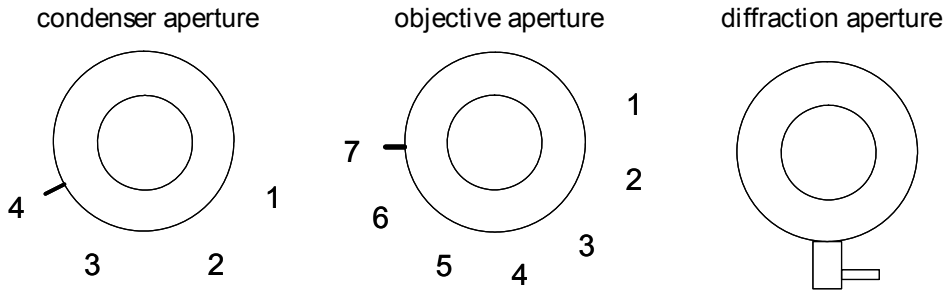
3. **Go** to the VACUUM STATUS page (from the HR-TEM page, press the VACUUM softkey). Check that the vacuum status is READY and that the IGP is less than 27. *If not, check with EMU staff.*

4. **Return** to the HR-TEM page (press READY).

5. **Check** that the High Tension indicator on the right side of the panel is lit. *If not, check with EMU staff.*

6. **Fill** the liquid nitrogen dewar and place it under the cold finger. You must wear safety glasses and protective gloves when handling liquid nitrogen.

7. **Ensure** that condenser aperture 4 and objective aperture 7 are selected and that the diffraction aperture is out (swing shift to the right).



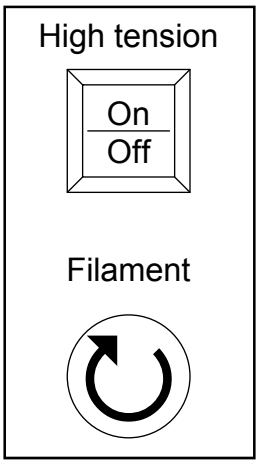
3. TURN THE FILAMENT UP

1. **Press** the MODES softkey and then the CONFIGURATION softkey to go to the CONFIGURATION page.

2. **Turn** the filament knob on the right-hand side of the display screen slowly clockwise (three or four clicks at a time).

   Note: If the voltage is increased too quickly, the kV value will freeze and you will need to seek assistance from EMU staff.

   Monitor the extraction voltage on the screen. When the voltage approaches its limit (~4.5 kV), you will hear a beep. Make sure you reach the maximum possible voltage by turning the knob a few more clicks. The voltage will usually reach 4.48 kV.



3. **Press** the READY button and then the HR-TEM softkey to return to the HR-TEM BRIGHT FIELD page.

4. LOAD THE SPECIMEN

There are two specimen holders available:

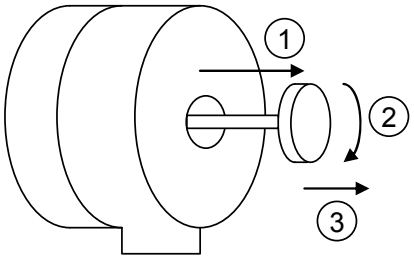
1) A single-tilt holder for conventional imaging and EDS analysis.

2) A low-background double tilt holder for crystallographic studies and specialised EDS analysis (see appendix a).

When you begin your cm200 session the single-tilt holder will normally be in the microscope.

Remove the single-tilt holder:

1. **CHECK** THAT THE GUN VALVE IS CLOSED. If not, close it by turning the switch slowly clockwise.
2. **Pull** the specimen holder out until it cannot be pulled any further (~8cm).
3. **Rotate** the holder clockwise as far as it will go (~120º).



4. **Pull** the holder gently out of the airlock. Take care not to knock the specimen holder against the airlock walls as it is released.
5. **Place** the specimen holder in the support. Do not touch the brass part of the specimen holder with your fingers.
6. **Lift** the specimen securing clamp using the special tool.
7. **Load** the specimen into the recessed ring, then gently lower the securing clamp using the special tool.
8. **Rotate** the specimen holder slightly and gently tap it to ensure that the specimen stays in place.

Insert the single-tilt holder:

1. **CHECK** THAT THE GUN VALVE IS CLOSED. If not close it by turning the switch slowly clockwise.
2. **Check** the IGP reading by going to the VACUUM STATUS page. The IGP reading should be less than 27. *If not, check with EMU staff.*

3. **Insert** the rod gently into the specimen airlock. Orient the rod so that the large key peg is at the 11 o'clock position. Push the rod into the airlock until you feel a stop and the red light on the airlock illuminates (this indicates that the airlock is being pumped). After the pump has started it is often useful to gently wiggle the rod, which allows it to be inserted another few millimetres.

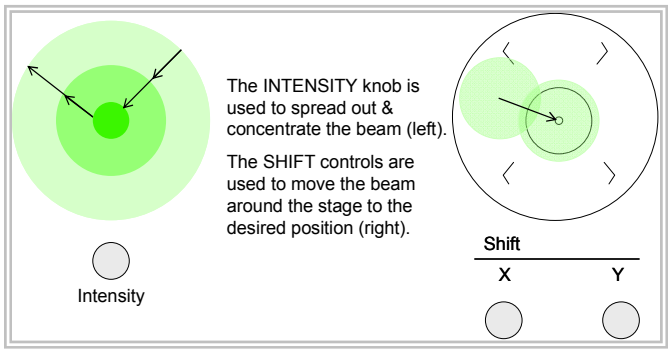
4. **Wait** until the red light has turned off.

5. **Rotate** the rod anti-clockwise (~120º) keeping a firm grip on the holder. The vacuum will 'grab' the rod when it gets to the 6 o'clock position.

6. **Guide** the rod as it enters the airlock. The key peg should slide into the slot on the airlock. Monitor any change in vacuum by noting the IGP value. (The IGP value may rise above 27 during rod insertion – it must be 27 before operation).

5. OBTAIN AN IMAGE

1. **Check** that the IGP reading is less than 27.
2. **Go** to the HR-TEM BRIGHT FIELD page (from the VACUUM page, press READY).
3. **Set** the magnification to ~500x.
4. **Open** the gun valve by turning the switch slowly anti-clockwise. The beam should appear on the viewing screen within a few seconds.
5. **If** the beam does not appear, check that the high tension indicator is still lit.
    - *If not, close the gun valve by turning the switch slowly clockwise and check with EMU staff.*
    - *If the high tension indicator is lit, check the magnification is ~500x and that the objective aperture is set to 7.*
    - *If you are examining an electro-polished specimen move the specimen around using the X-Y joystick until you find the hole*
6. **Centre** the beam using the X and Y shift controls.
7. **Spread** the beam over the screen by turning the INTENSITY knob.
8. **Locate** the area of interest on the specimen and place it in the centre of the stage using the X-Y joystick.


The INTENSITY knob is used to spread out & concentrate the beam (left).
The SHIFT controls are used to move the beam around the stage to the desired position (right).
Intensity
Shift
X
Y

6. SET THE EUCENTRIC HEIGHT

1. **Increase** the magnification to 5 -10K.
2. **Spread** the beam over the screen by turning the INTENSITY knob.
3. **Move** the beam to the centre of the screen using the X and Y SHIFT controls.
4. **Move** the specimen (X-Y joystick) so that a recognizable feature is in the centre of the screen.
5. **Go** to the COMPUSTAGE page.
6. **Press** the A-WOBBLER softkey. This causes the specimen to tilt continuously from +15º to -15º. If the specimen is not at eucentric height the image will move back and forth on the screen.
7. **Adjust** the height of the specimen using the Z joystick until the movement of the image is minimized.
8. **Press** the A-WOBBLER softkey to stop the specimen tilting.

7. FOCUS THE IMAGE

1. **Check** that the image is in focus using the FOCUS control. FOCUS STEP (sensitivity of focus control) can be adjusted using the inner control knob.

    - Use the small screen (lift by using the rear lever on the left-hand side of the column) and the binocular microscope on the front of the column to attain the best focus.
    - Check that the binoculars are focused by inserting the pointer (right hand side of the column; turn to insert) and adjust the eyepieces until the image of the pointer is focused. To remove the pointer pull it out and turn it to lock.
    - When focusing, start with a large focus step (5 or 6) and get the best focus you can before progressively reducing the focus step to achieve fine focus (focus step 2 or 3).

8. MOVE AROUND THE SPECIMEN

- The X-Y joystick controls x-y movement of the specimen. The Z joystick should be adjusted only when eucentric height is being set.

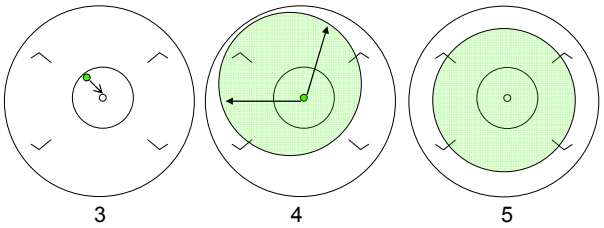
- The stage position can be monitored on the HR-TEM page or the COMPUSTAGE page. On the COMPUSTAGE page, stage positions can be stored and recalled (see section 1).

- Specimen tilt.

    - a-tilt is available on both the single and double tilt holder. The specimen is tilted by turning the TILT knob either clockwise or anti-clockwise depending on the desired direction of the tilt. The extent to which the knob is turned determines the tilt speed.
    - B-tilt is available only with the double-tilt holder. The direction and speed of the tilt are controlled by the HOLDER knob. The tilt is controlled by the foot pedal
    - Reset the specimen to 0º tilt by pressing RESET AB on the COMPUSTAGE page.

9. CHECK THE MINOR ALIGNMENTS

The minor alignments should be checked at the start of each session, and may be adjusted at any time. They should be performed whenever the condenser aperture is changed.



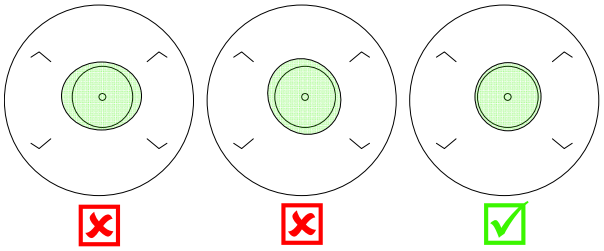
Condenser aperture alignment

The aim of this alignment is to make the beam expand and contract concentrically.

1. **Check** that the objective aperture is out (position 7).
2. **Focus** the image.
3. **Condense** the beam to a fine spot using the intensity knob.
4. **Centre** the spot on the screen using the X and Y shift knobs.
5. **Expand** the spot using the INTENSITY knob until it is ~10cm in diameter.
6. **Centre** the spot on the screen using the condenser aperture drives.
7. **Repeat** from step 2 until the beam expands concentrically from the centre of the screen.

Condenser stigmation alignment

The aim of this alignment is to make the beam as circular as possible.



1. **Check** that the objective aperture is out (position 7).
2. **Set** the magnification to ~50K.
3. **Focus** the image.
4. **Press** the STIG button on the right hand side of the console. The LED will illuminate and the STIGMATOR CONTROL page will be displayed.
5. **Select** the COND softkey.

6. **Condense** the beam to a spot about 3cm in diameter using the INTENSITY knob.

7. **Adjust** the beam so that it is as circular as possible using the X and Y MULTIFUNCTION knobs on the right hand side of the console.

8. **Press** the STIG button to leave this function.

Pivot point alignment

If the pivot points are aligned, focussing at high magnifications is easier

1. **Check** that the objective aperture is out (position 7).

2. **Set** the magnification to ~50K.

3. **Focus** the image.

4. **Condense** the beam to a small spot.

5. **Press** the ALIGN button on the right hand side of the console. The LED will illuminate and the ALIGNMENT SELECTION page will be displayed.

6. **Press** the PIVOT POINT X softkey. You should see two spots on the screen. (If the spots are not visible you may need to reduce the magnification).

7. **Use** the MULTIFUNCTION X and Y knobs on the right hand side of the console to make the two spots overlap. (Keep the spots in view by adjusting the X and Y SHIFT controls, if necessary).

8. **Press** the PIVOT POINT Y softkey. You should see two spots on the screen. (If the spots are not visible you may need to reduce the magnification).

9. **Use** the MULTIFUNCTION X and Y knobs on the right hand side of the console to make the two spots overlap. (Keep the spots in view by adjusting the X and Y SHIFT controls, if necessary).

10. **Press** the ALIGN button to leave this function.

10. INSERT AN OBJECTIVE APERTURE

There are 4 objective apertures available. The largest aperture (1) produces the least image contrast; the smallest (4) produces the most contrast. Positions 5, 6 and 7 do not have an aperture.

1. **Set** magnification to 5-10K.

2. **Condense** the beam to a fine spot using the INTENSITY knob.

3. **Press** the D button on the right hand side of the console. This will produce a diffraction pattern on the screen.

4. **Turn** the objective aperture control counter clockwise to select the desired aperture.

5. **Centre** the aperture (the dim circle) over the bright spot using the aperture alignment knobs. *Be careful! The amount of movement required is very small.*

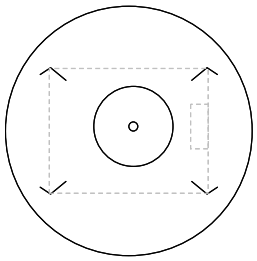
6. **Press** the D button again to go back to a normal (bright-field) image.

7. **Spread** the beam over the screen using the INTENSITY knob.

11. RECORD AN IMAGE ON PHOTOGRAPHIC FILM

1. **CHECK** THAT THE IMAGE SELECTOR SWITCH IS SET TO CM.

2. **Align** the area of interest on the centre of the screen. The corners of the negative are marked by the middle spot of each group of three spots. The data bar (including the scale bar and exposure number) occupies the far right of the image, so make sure that your primary area of interest is not in this region.



3. **Check** that the image is in focus using the FOCUS control. FOCUS STEP (sensitivity of focus control) can be adjusted using the inner control knob.

    - Use the small screen and the binocular microscope on the front of the column to attain the best focus.

4. **Spread** the beam so that it covers the whole screen. The exposure time is set automatically.

5. **Note** the exposure number. This number is the number of the next negative to be used and will appear on the negative when it is developed.

6. **Lift** the main screen into the vertical position using the front lever on the left-hand side of the column. The light on the EXPOSURE button on the left-hand side of the column will turn on.

    - If the EXPOSURE light does not illuminate, check that there is still film in the camera (stock) and that the exposure time is not too long. The EXPOSURE light will not illuminate when the rotary pump is operating – wait until it stops to take your image.

7. **Press** the exposure button. All of the console lights will turn off and the film will be exposed.

8. **Wait** until the console lights turn back on and then lower the screen using the lever on the left-hand side of the column. The EXPOSURE light will turn off when the stage is lowered.

12. RECORD A DIGITAL IMAGE

1. **CHECK** THAT THE IMAGE SELECTOR SWITCH IS SET TO CCD.

2. **Align** the image in the centre of the screen. The digital camera captures the area shown on the small screen (lift by using the rear lever on the left-hand side of the column). The digital image will be rotated by 90º.

3. **Check** that the image is in focus using the FOCUS control. FOCUS STEP (sensitivity of focus control) can be adjusted using the inner control knob.

    - Use the small screen and the binocular microscope on the front of the column to attain the best focus.

4. **Spread** the beam so that it covers the whole screen.

5. **Start** ANALYSIS on the PC by clicking on START>PROGRAMS>AnalySIS> AnalySIS.

6. **Lift** the main screen into the vertical position using the front lever on the left-hand side of the column.

7. **Click** on the ACQUIRE icon .

    - This image is 'live' and will be refreshed every few seconds.

    - The camera control icon controls the brightness/contrast. Adjust the exposure time so that the intensity histogram is normally centred (usually 1000ms).

8. **Click** on the SNAPSHOT icon to capture the image.

9. **Click** on the SCALEBAR icon to add a scale bar.

10. **Save** the image into your folder on the network (m:\images\cm200\your user name).

11. **Lower** the stage to continue imaging.

13. EXCHANGE SPECIMENS

1. **Reduce** the magnification to ~500x.
2. **Remove** the objective aperture by turning the aperture control fully clockwise so that it is in position 7.
3. **CLOSE THE GUN VALVE** by turning the control knob slowly clockwise.
4. **Ensure** that the stage is at 0º tilt by going to the COMPUSTAGE page and pressing the RESET AB softkey.
5. **Pull** the specimen holder out until it cannot be pulled any further (~8cm).
6. **Rotate** the specimen holder ~120º clockwise.
7. **Pull** the specimen holder gently out of the airlock.
8. **Place** the specimen holder in the support.
9. **Lift** the specimen securing clamp using the special tool.
10. **Remove** the specimen.
11. I**nsert** the new specimen into the holder.
12. **Lower** the specimen securing clamp using the special tool.
13. I**nsert** the specimen holder into the column as described in section 4.

14. END THE SESSION

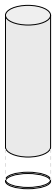
1. **Remove** the specimen as described in section 13.
2. **Insert** the single tilt specimen holder into the column as described in section 4.
3. **Go to** the CONFIGURATION page by pressing MODES then CONFIGURATION.
4. **Press** the STANDBY softkey to put the microscope into standby mode. Check that the filament emission automatically reduces to ~2.29kV.
5. **Clear** any stored stage positions by going to the COMPUSTAGE page and pressing CLEAR ALL twice.
6. **Cover** the TEM windows.
7. **Exit** from any programs that you have been running on the PC.
8. **Log out** of the EMU booking system.

Appendix a: Using the double tilt holder

The double-tilt holder may be used by advanced users for crystallographic studies and/or specialised EDS analysis.

If you would like to use the double-tilt holder, contact a member of EMU staff for training.

1. **Close the gun valve** and **remove** the single-tilt holder as described on pX.
2. **Rest** the double-tilt holder in the clear plastic support.
3. **Unscrew** and remove the beryllium ring using the tool provided (kept in a plastic, snap-top tube).



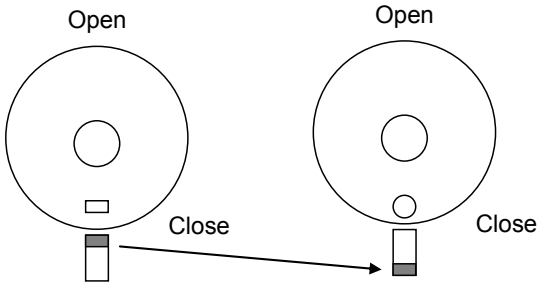
4. **Load** your specimen into the circular recess in the holder.
5. ***Gently*** and carefully **screw** the beryllium ring into place to secure the sample, using the tool provided.

AVOID CROSS-THREADING & DO NOT OVER-TIGHTEN THE RING

6. **Rotate** the specimen holder slightly and gently tap it to ensure that the specimen remains in place.
7. **Check** the IGP reading by going to the VACUUM STATUS page. The reading should be less than 27.

If not, check with EMU staff

8. **Lower** the shift on the airlock so that the circular slot is open.



9. **Orient** the rod so that the large key peg is at about 11 o'clock. **Insert** the rod gently into the specimen airlock until the red light on the airlock illuminates (this indicates that the airlock is being pumped). After the pump has started it is often useful to gently wiggle the rod, which allows it to be inserted by another centimetre or so.

DO NOT INSERT THE ROD FURTHER UNTIL THE RED LIGHT HAS TURNED OFF.

10. The computer system will automatically sense that a double-tilt holder is being inserted and will ask you to specify which specimen holder you are using. On the computer interface, **select** PHILIPS DOUBLE TILT and **press** READY.

11. As instructed on the screen, **plug** in the double-tilt holder cable to the right side of the airlock and **press** READY.

12. **Check** that the red light is off.

13. **Rotate** the rod ~120° anti-clockwise ↺, keeping a firm grip on the holder. (The vacuum will 'grab' the rod when it gets to about 6 o'clock.)

14. **Guide** the rod as it enters the airlock. The key peg should slide into the slot on the airlock. Monitor any change in vacuum by noting the IGP value. (IGP may rise above 27 during rod insertion – **it must be below 27 before operation**).

Appendix b: Diffraction patterns

1. Select the required field of view with the sample at the appropriate tilt angle.
2. Insert a selected area diffraction aperture (if required) by turning the swing shift to the left. Align the aperture over the area of interest.
3. Remove the objective aperture from the beam.
4. Press the D button.
5. Adjust magnification (camera length), focus and intensity to optimise the pattern.
6. Centre the pattern on the screen with the MULTIFUNCTION X and Y knobs.
7. Record the pattern.
    - Check that the image selector switch is set to CM.
    - Select TEM CAMERA.
    - Adjust MAN TIME to set the exposure time. For most patterns 1-2 seconds is appropriate. (Exposure time is best determined by running a series of test exposures.)
    - Note the exposure number.
    - Lift the main screen.
    - Press the exposure button.
    - Wait until the console lights turn back on and then lower the main screen.
8. Insert and align the objective aperture.
9. Remove the selected area diffraction aperture.
10. Press the D button to leave diffraction mode.

Appendix c: X-ray analysis

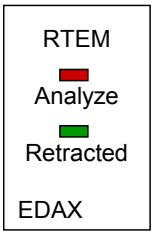
SEE EMU STAFF BEFORE ATTEMPTING X-RAY ANALYSIS FOR THE FIRST TIME.

The Philips CM200 is equipped with an EDAX r-TEM system to perform energy-dispersive x-ray spectroscopy (EDS). We use a Sapphire Si(Li) EDAX detector with 30mm2 active area. This is used to acquire and analyse chemical spectra from the TEM sample. The software programs used to run the analyses operate in a Windows™ environment and many of the functions of the software are similar to other Windows-based programs.

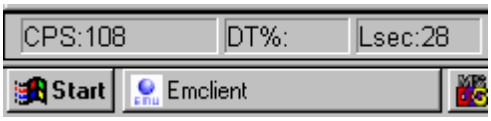
These instructions are intended as a basic guide. For advanced software features, please consult the mDX or iDX Reference Manual available from EMU staff.

Getting Started

1. **Start** the EDS software by double-clicking on the mDX icon.
2. **Locate** the region of interest in the centre of the TEM viewing screen.
3. **Remove** the objective aperture.
4. **Insert** condenser aperture 3. **Align** the aperture (see p.23).
5. **Tilt** the specimen toward the detector by increasing the α-tilt to approximately +15°.
6. **Reduce** the spot size to spot size 4. You may need to adjust the spot size again later.
7. **Position** the beam over the area of interest using the INTENSITY and BEAM SHIFT knobs.
8. **Insert** the EDS detector by pressing the red ANALYZE button on the RTEM hardware control box.



9. **Check** the COUNT RATE (bottom left corner of the computer screen). It should be between approximately 1000 counts per second (cps) and 4000 cps.



- If the count rate is much too high, the detector will retract automatically. The green RETRACTED button will flash. **Press** the RETRACTED button to stop the flashing. **Diagnose** the cause of the high count rate (e.g., objective aperture left in or Cu grid in

- If the count rate is over 4000cps but the detector does not automatically retract, **try decreasing** the spot size to reach 1000-4000cps.

- If the count rate is too low (<~200cps), **increase** the spot size, re-position the beam and re-acquire.

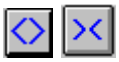
Acquiring a Spectrum

1. **Click** on the clock icon in the top left corner of the mDX menu bar to start acquiring a spectrum. The icon will turn yellow when activated. The spectrum will automatically acquire for 100 seconds. To stop acquisition at any time, click on the clock icon again (it will turn grey).

2. **Check** the count rate and adjust if necessary.

Modify Spectrum Appearance

1. The **scale** of the spectrum can be manipulated by holding down the left mouse button and moving the mouse left, right, up or down. The spectrum can be expanded or contracted by pressing the appropriate buttons on the toolbar:

   - Expand/contract horizontally: 

   - Expand/contract vertically:

   - Press the house icon to restore the full spectrum.

2. **Add text to the spectrum** by selecting ADD TEXT from the EDIT menu. **Click** on the spectrum at the point where the text is to be added and **enter** the text in the box that appears. **Press** ENTER to place the text on the spectrum. The text can be edited or removed at any time by re-selecting ADD TEXT from the EDIT menu and then clicking on the text.

3. **Add text to the spectrum header** by selecting LABEL from the EDIT menu, **enter** a label in the box and **press** OK.

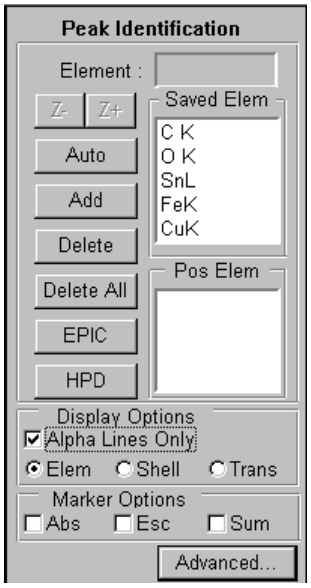
4. **Erase a spectrum** by pressing the paint-roller icon in the top left corner of the screen.

Saving Spectral Data

1. **Save** files to your username folder under M:\ > IMAGES > CM200.
2. Spectrum files can be saved in .spc, .tif, .csv or .bmp format.
   - .spc files can only be opened by the mDX software. It is good practice to save these files in case you need to manipulate them in future.
   - .tif and .bmp files save the spectrum as a picture that can be opened in most imaging software programs.
   - .csv files save a spectrum trace as x and y data points. This type of file allows the user to re-graph the data in a spreadsheet program such as Microsoft Excel.

Qualitative Elemental Analysis

The EDS system we use is almost exclusively used for the acquisition of qualitative or semi-quantitative data. For accurate quantitative data analysis, users should consider using the microprobe or performing mass spectrometric analysis.

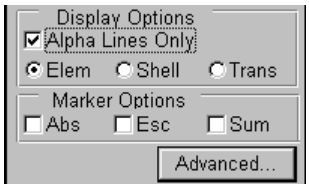


1. The series of icons in the top right corner of the mDX screen are used for interpreting the spectral data. **Select** the PEAK IDENTIFICATION icon to open the PEAK IDENTIFICATION dialogue box on the right side of the screen.
2. **Click** on the AUTO button. This will automatically identify and label the main peaks. The list of identified elements will also appear in the SAVED ELEM box.
3. **Check** that each automatically identified peak is definitely associated with a particular element by clicking on the element (e.g., FeK) in the SAVED ELEM box. This will display all of the peak positions associated with that element. If a peak has been incorrectly identified, **select** that element in the SAVED ELEM box and **click** on the DELETE box to remove it from the list.
4. **Manually identify** a peak by clicking on the peak itself. The possible elements associated with that energy value (keV) will be displayed in the POS ELEM box. **Click** on a possible element in the POS ELEM box to display

all of the peak locations associated with that element. When you identify an element, **click** on ADD to add the element to the SAVED ELEM box.

Adjust the Display Characteristics

1. Element labels can be removed individually from the spectrum (and SAVED ELEM box) by **highlighting** them and **clicking** on the DELETE button. Clicking on the DELETE ALL button will remove all peak labels and will clear the SAVED ELEM box.**Select** the ALPHA LINES ONLY box to remove labels from all peaks except the α peaks.



2. **Select** ELEM to label peaks by element (e.g., Cu or Sn); SHELL to label peaks by their electron shell level (e.g., CuK or SnL); or TRANS to label peaks by their transition type (e.g., CuKa or SnLb).

3. **Select** EPIC to bring up a periodic table with x-ray energies to assist in peak identification. Select an element and click on ENERGY TABLE to display peak energies.

Regions of Interest (ROI)

Energy ranges (regions of interest) representing certain element peaks can be selected for use in elemental mapping (see p.44). Peaks falling within the selected ROI will be shaded (default colour is grey).

To remove an ROI label, select the the ROI icon and click on the DELETE button. Select DELETE ALL to remove all ROI labels at once.

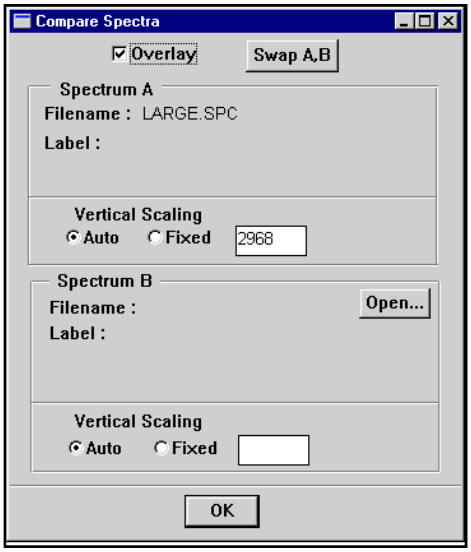
Comparing Two Spectra

1. **Click** on VIEW on the menu bar and select COMPARE. A COMPARE SPECTRA window will appear showing the name of the last spectrum acquired as **Spectrum A**.

2. **Click** on the OVERLAY box.

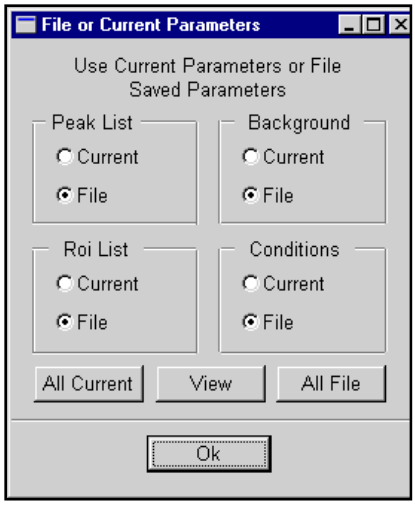
3. **Open** a second, saved spectrum by clicking on OPEN. **Select** your second spectrum for comparison (**Spectrum B**).

4. **Click** on OK. The two spectra will be overlayed for comparison.



Opening a Saved Spectrum

1. To **open** a previously saved spectrum, **click** on FILE > OPEN, **locate** your folder and file and **click** on OK.



2. A FILE or CURRENT PARAMETERS dialogue box will appear with a selection of display options. For most applications, **ensure** that all the FILE options are selected. This will display the spectrum in the same format as it was saved.

3. **Select** the VIEW option to look at the parameters stored with the saved spectrum.

4. **Click** OK to open the spectrum.

Semi-Quantitative Analysis

The mDX program's QUANTIFY function allows the user to perform semi-quantitative elemental analysis of a sample. Proper quantitative analyses can be conducted but require replicate analyses and the careful preparation and use of standards in order to assess the accuracy and precision of the results.

Using mDX, first identify the elements to be quantified, subtract the background, and select the appropriate set of correction parameters. The following description is generic and should be modified depending on the nature of the quantification to be performed. Consult the reference manual and EMU staff for assistance.

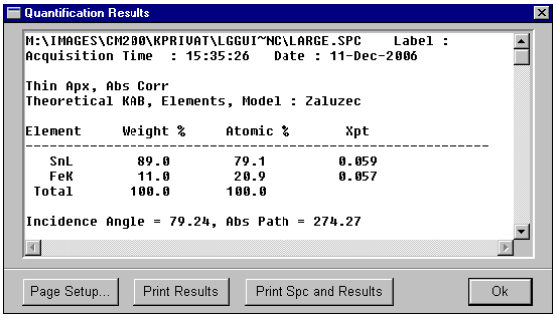
1. **Click** on the QUANT icon in the top right corner of the computer screen. The QUANTIFY control panel will appear.

2. **Click** on the Z List button. A Z LIST dialogue box will appear, listing the elements to be quantified.

3. **Select** or de-select the elements to be quantified by clicking on them. **Click** OK.

4. **Click** on the Background : Auto button. A BACKGROUND display panel will appear.

5. **Select** SET to generate a list of elements identified using the qualitative analysis in the AUTO field. These elements are used to determine the background radiation.


Quantification Results
M:\IMAGES\CM200\KPRIVAT\LGGUI~NC\LARGE.SPC Label :
Acquisition Time : 15:35:26 Date : 11-Dec-2006
Thin Apx, Abs Corr
Theoretical KAB, Elements, Model : Zaluzec
Element Weight % Atomic % Xpt
SnL 89.0 79.1 0.059
FeK 11.0 20.9 0.057
Total 100.0 100.0
Incidence Angle = 79.24, Abs Path = 274.27
Page Setup...
Print Results
Print Spc and Results
Ok

6. **Click** on the – (subtract) button in the SUBTRACT field to remove the background. **Select** UNDO to restore the background to the spectrum.

7. **Click** on the QUANT icon .

8. **Click** on the Type : Oxides button to open the TYPE control panel. Normally, the ELEMENTS option is selected, which excludes oxides from the quantification calculations. If oxygen peak intensity has been measured, ELEMENTS should be selected. For analysis of oxide mixtures (where the oxygen peak intensity has not been measured), select OXIDES.

9. **Click** on the K Factor : Theoretical button to select the type of quantification calculation to be used. A KAB FACTORS field appears in the QUANTIFICATION panel. In most cases, select THEO.

If you need to define your own correction factors/method, see Paul Munroe.

10. **Click** on the Corrections : Abs Corr button to select the correction factors applied. A correction factor will appear in the QUANTIFICATION panel. For most applications, **select** either INTEN only or THIN, INTEN and ABS, depending on the type of correction required. THIN denotes thin approximation calculations, INTEN denotes intensity calculations, ABS denotes absorption correction calculations and FLUORESENCE denotes absorption correction calculations with fluorescence.

11. **Click** on the Quant button to perform the quantification calculations. If you have elected to do an absorption correction, an ABS CONDITIONS window will appear. **Enter** the nominal specimen thickness (typically ~200nm), a-tilt value and the nominal specimen density and click OK.

12. The results will be displayed in a QUANTIFICATION RESULTS window. **Save** results by clicking on the Save button in the RESULTS panel.

Ending the EDS Session

1. **Remove** the EDS detector by pressing the green RETRACTED button on the RTEM hardware control box to the right of the column.

2. **Press** the AB RESET softkey on the COMPUSTAGE page to return the specimen to 0° tilt.

3. **Insert** and **align** condenser aperture 4 (see p.15).

4. **Exit** the mDX program.

5. **End** the session as described in section 14.

Appendix d: X-ray mapping/line scans

X-ray maps can be made using the iDX software installed on the computer attached to the CM200. A map is a two-dimensional representation of elemental distribution in a sample. A map is made by obtaining an STEM image, transferring the STEM image to the PC, and acquiring spectra across the selected sample area on a pixel-by-pixel basis.

SEE EMU STAFF BEFORE ATTEMPTING THIS FOR THE FIRST TIME.

Getting Started

1. **Check** that the image selector switch is set to CM.
2. **Obtain** a well-focused image on the TEM viewing screen.
3. **Tilt** the specimen so that an α-tilt of approximately +15° is achieved.
4. **Insert** and **align** condenser aperture 3 (see p.15).
5. **Align** the condenser stigmator (see p.15).
6. **Remove** the objective aperture.
7. **Adjust** the magnification to 10,000-20,000x.
8. **Condense** the beam to a small spot, **centre** the beam using the X and Y shift controls, and **press** the D button to obtain a diffraction pattern.

Obtaining a STEM Image

1. **Press** the MODES softkey and then **press** the SCAN BF-DF softkey *twice* to view the SCANNING page.
2. **Lift** the TEM stage and a STEM image should be displayed on the two monitors on the top of the microscope console. The bright-field image should be visible on the left monitor, and a dark-field image on the right. (The magnification may need to be reduced to ~20,000x using the magnification control.)
3. **Ensure** that the bright-field image is active. Two LEDs below the screen will be illuminated when the screen is active. **Use** the DETECTOR CONTROL button in the SIGNAL CONTROL area of the STEM controls to toggle between the screens.
4. **Centre** the beam.
    I. **Press** ALIGN go to the ALIGNMENT SELECTION page.

II. **Press** the DET ALIGNM softkey

III. **Centre** the illumination on the bright field image using the X and Y MULTIFUNCTION controls. The magnification may need to be reduced in order to see the illumination spot. If magnification is changed, the DET ALIGNM softkey must be pressed again in order to activate the X and Y MULTIFUNCTION knobs.

IV. **Press** ALIGN to exit the ALIGNMENT SELECTION page.

5. **Optimise** the image.

   - **Adjust** the image position, magnification and focus using the standard TEM controls. The STEM image may be rotated by turning the MULTIFUNCTION X knob.
   - **Adjust** the brightness and contrast of the image using the BRIGHTNESS and CONTRAST controls on the STEM control panel.
   - **Adjust** the SCAN RATE at any time by selecting the appropriate button: SLOW or FAST.

Collecting the Electron Image

1. **Enable** external control of the microscope by pressing the EXP 3 button on the PHOTO SCAN RATE section of the STEM control panel.
2. **Open** the iDX program.
3. **Click** on the icon in

the toolbar to acquire an electron image (or select COLLECT > ELECTRON IMAGE). The image will be refreshed continuously until the icon is pressed again.
4. **Adjust** image parameters.

   I. Click on the

icon.

   II. Select the matrix size.

   III. Enter the magnification.

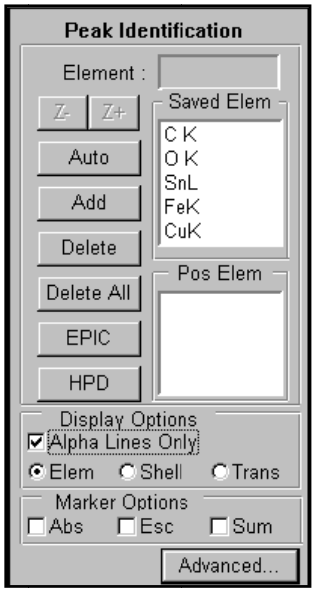
   IV. Display a micron bar by selecting MICRON BAR from the DISPLAY menu.

   V. Adjust the brightness and contrast of the image using the BRIGHTNESS and CONTRAST controls on the STEM control panel.

Select regions of interest

1. **Insert** the EDS detector by pressing the red ANALYZE button on the RTEM hardware control box.

2. **Select** SPECTRUM from the COLLECT menu. A spectrum should begin to acquire. Acquisition can be started manually by clicking on the button on the spectrum toolbar. Stop the acquisition by pressing the button again.



3. **Select** the PEAK IDENTIFICATION icon .

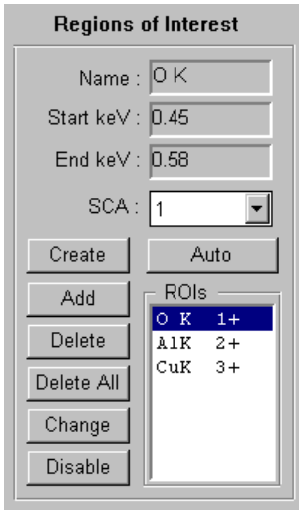
4. **Click** on the AUTO button. This will automatically identify and label the main peaks. The list of identified elements will also appear in the SAVED ELEM box.

5. **Check** that each automatically identified peak is definitely associated with a particular element by clicking on the element (e.g., FeK) in the SAVED ELEM box. This will display all the peak positions associated with that element. If a peak has been incorrectly identified, **select** that element in the SAVED ELEM box and **click** on the DELETE box to remove it from the list.

6. **Manually identify** a peak by clicking on the peak itself. The possible elements associated with that energy value (keV) will be displayed in the POS ELEM box. **Click** on a possible element in the POS ELEM box to display all the peak locations associated with that element. When you identify an element, **click** on ADD to add the element to the SAVED ELEM box.

7. **Click** on the button to open the REGIONS OF INTEREST (ROI) panel on the right side of the screen. **Delete** any previously saved ROI by clicking on DELETE ALL.

8. **Click** on the AUTO button. This will create a ROI energy range for all of the elements in the PEAK IDENTIFICATION panel. An individual map of each of the elements selected in the ROI list will be acquired. If you do not want to map all of the elements listed, **remove** the unwanted element(s) from the list by selecting the element and clicking on DELETE.



Acquiring a map

1. **Click** on the button to open the X-MAPS PARAMETERS panel.
2. **Adjust** the resolution of the image/maps by changing the size of the MATRIX (# data points/pixels) and the DWELL TIME.
    - Usually a 128 x 100 collection matrix with a 10ms dwell time is sufficient to obtain at least a preliminary idea of the results.
    - A 256 x 200 collection matrix with a 10ms dwell time usually takes ~20 minutes, depending on the number of elements to be mapped.
    - Increasing the dwell time and the matrix size will increase the resolution, but will also increase the acquisition time.
3. **Click** on the button to start the acquisition (or select X-RAY MAPS from the COLLECT menu).
    - A prompt will ask for a filename. Filenames must be ≤4 characters long. **Enter** an appropriate filename and select your folder (under M: > Images > CM200). Files will be saved as bitmaps; file size depends on the size of the collection matrix. **Click** OK.
    - A prompt box will display the collection parameters you have chosen. **Click** on YES to start the map acquisition.
    - To **stop** the acquisition at any time, click on the button again (or **select** STOP from the COLLECT menu).
    - The maps are automatically saved when acquisition is complete.

Acquiring a line scan

1. **Click** on the icon to open the LINE SCAN PARAMETERS panel.
2. **Click** the icon to activate the line drawing function.
3. **Click** and drag on the image to draw a line. To redraw the line, click on the icon again.
4. **Adjust** the parameters of the line scan.
    - **Select** the number of points.

- **Select** the dwell time.
- The estimated time for the line scan is displayed in the status bar.

5. **Click** on the icon to start the acquisition.
   - A prompt will ask for a file name. Select your folder (under M:>images>cm200). Files will be saved as .csv files. They may be imported into MS Excel spreadsheets.

Acquiring further maps/line scans

1. **Press** the SLOW (or FAST) button on the VIEWING SCAN RATE panel to return to STEM mode.
2. **Adjust** the image as required (position, brightness, contrast, magnification, etc.).
3. **Continue** as described from ***Collecting the Electron Image*** (p.33).

Ending the Mapping Session

1. **Remove** the detector by pressing the green RETRACTED button on the RTEM hardware control box to the right of the column.
2. **Press** the SLOW button on the VIEWING SCAN RATE panel to return to STEM mode.
3. **Lower** the viewing screen on the TEM.
4. **Press** the MODES softkey and **press** HR-TEM *twice* to return to the HR-TEM main page.
5. **Go** to the COMPUSTAGE page and **press** the RESET AB softkey to return the specimen to 0° tilt.
6. **Insert** and align condenser aperture 4 (see p. 15).
7. **Quit** the iDX software.
8. **Log out** of the EMU system and **turn off** the monitor.